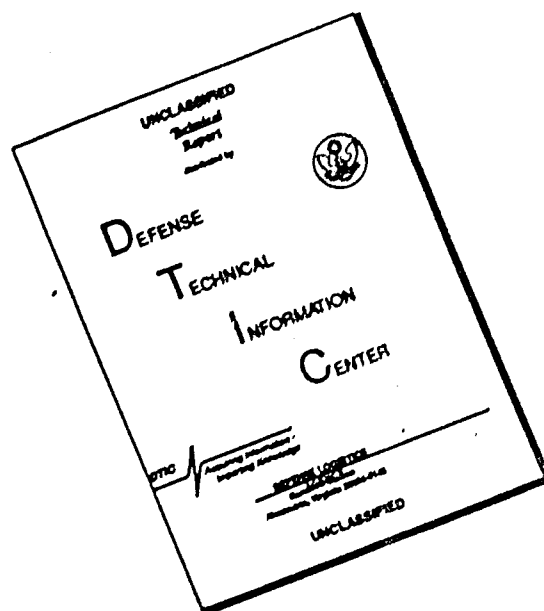


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DEVELOPMENT AND APPLICATION
OF A THEORY FOR PLASTIC DEFORMATION
OF CEMENTED ALLOYS

Task 1

ZONE REFINING OF CEMENTED TUNGSTEN-BASE ALLOYS

Technical Report WAL TR 372/32-1-T1

by

N.M. Parikh

October 27, 1961

Quarterly Report
July 1, 1961 - September 30, 1961

Contract No. DA-11-022-505-ORD-3092
Chicago Ordnance District

ORD Project TBL-002
D/A Project 5B93-32-003
Watertown Arsenal
Watertown 72, Massachusetts

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ORD Project TB4-002
D/A Project 5B93-32-002
Watertown Arsenal
Watertown 72, Massachusetts

WAL 372/32-1-T1

ZONE REFINING OF CEMENTED
TUNGSTEN-BASE ALLOYS

ABSTRACT

This program concerns the development of a process for fabrication of tungsten sheet using tungsten-nickel-iron alloys as a starting material. Initial attempts in scaling up the process were successful. Sintering, warm rolling and zone refining tungsten-nickel alloys presented no difficulties. Eight inch wide hot rolled tungsten sheets were made by this process.

ZONE REFINING OF CEMENTED
TUNGSTEN-BASE ALLOYS

I. INTRODUCTION

This is the first quarterly report under the revised contract for ARF Project No. 2182, entitled "Development and Application of a Theory for Plastic Deformation of Cemented Alloys." This report (WAL TR 372/32-1-T1) covers the work done under Section (a)(8)b, entitled "Zone Refining of Cemented Tungsten-Base Alloys," from July 1 to September 30, 1961.

The current methods of manufacture of sheet tungsten are fraught with severe limitations. The sheets available to date—usually made in small lots—are made by hot breakdown of arc-cast or sintered billets followed by hot rolling at 1400° to 1700°C. This is then followed by warm rolling at temperatures below 1400° down to about 1100°C. Each one of the steps involved is difficult and usually results in substantial scrap losses. The technique of arc casting is satisfactory for billets up to 4 or 5 inches in diameter. Beyond this size, thermal cracking is understood to be a major problem. The powder metallurgy approach used by some manufacturers involves compacting powders and sintering the compacts from 2500°-3000°C. The temperature of sintering is very critical in that the higher temperatures yield a higher density product, and it has been established that a sintered density of 90 per cent of theoretical is marginal—but not a safe one—insofar as subsequent hot-rolling is concerned. These requirements for high-density billets impose severe burdens on existing sintering facilities; moreover, the capability and the size of sintering furnaces available is not large enough to make billets of any appreciable width. The subsequent operations which involve hot breakdown by forging or direct rolling of sintered sheet bars usually result in heavy scrap losses and impose severe

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wear and tear on the equipment. It is agreed in the industry that a scrap loss of 40-60 per cent during hot breakdown and rolling is not unusual. One novel technique which shows some promise is that of centrifugal casting of tungsten pipes up to 12 inches in diameter and 1 inch wall thickness followed by slitting, flattening at 1000°F, and hot-rolling to sheet. This technique has been used successfully for molybdenum, but for tungsten it appears that considerable work will have to be done before one can produce sound castings and sheet by this method. Skull casting of tungsten is yet another method of making thin-walled pipe but is far from reaching even a pilot-scale operation.

Those who make tungsten by powder metallurgy claim that two major difficulties must be overcome if one is to make wider and cheaper sheets:

- (1) Lower the sintering temperatures below 1800°C and
- (2) Eliminate the costly and difficult operation of intermediate breakdown. In other words one should obtain a billet as close to the finished thickness of the sheet that is desirable.

This particular program was initiated, therefore, with the above-mentioned objectives in mind; the work done so far indicates that these difficulties can be overcome by an entirely different route adopted in this program as described below.

It is well known that tungsten can be sintered to almost full density with as little as 0.5 per cent nickel. The mechanism of sintering in such alloys is that of liquid-phase sintering, and the resultant microstructure is two-phase wherein the tungsten grains are embedded in a semi-continuous nickel-rich matrix. In the previous work on the subject contract, it was established that high-tungsten alloys which were sintered with nickel and iron exhibited a marked degree of room-temperature ductility. For instance, an alloy of tungsten with 2.8 per cent nickel and 1.2 per cent iron can be cold rolled about 50 per cent. The main difficulty with these alloys is that the

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presence of the relatively low-melting nickel-rich matrix renders them unsuitable for high-temperature use. Under modification No. 1 of the subject contract, we had established that the low-melting, nickel-rich phase can be almost completely removed from the alloy by zone refining at reasonable temperatures ($\sim 1800^{\circ}\text{C}$). The product obtained by this method was almost pure tungsten. The high-temperature properties of tungsten thus prepared were comparable to those of tungsten prepared in a conventional manner as described in the early paragraphs of this section. Hence, it was decided to scale up this method for fabrication of sheet tungsten. The ultimate objective is to develop this process to a degree which is satisfactory for fabrication of as wide a sheet of tungsten as possible.

For successful execution of this program, it was decided to use the industrial facility of the Wah Chang Corporation in Albany, Oregon, for some phases of the program.

II. EXPERIMENTAL TECHNIQUES

The major portion of the effort during this report period was devoted towards a first series of experiments, both at Wah Chang Corporation and at ARF, and evaluation and testing of the finished sheets.

The raw materials used in the program were tungsten powder (2-4 microns) from Wah Chang Corporation, iron powder (-325 mesh) from Glidden Company, and nickel powder (-325 mesh) from International Nickel Company. The powders were blended in a proportion of 96 per cent tungsten, 2.8 per cent nickel and 1.2 per cent iron. The blended powders were then compacted into two slabs $10 \times 1.5 \times 0.75$ inch in a die at about 35,000 psi. The slabs were subsequently sintered in hydrogen at 1530°C for two hours and warm cross-rolled about 40 per cent at 750°F without any sign of edge cracking. The bars were then suspended vertically in an electron-beam zone refining furnace with 18-inch electrodes. The zone refining was accomplished in a

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vacuum of the order of 10^{-5} mm of Hg at 1900°C, and the bars were moved upwards at 2 inches per hour. Due to the geometry of the samples, the zone-refined bars were only about 8 x 2.5 x 0.5 in. after trimming. These bars were then hot cross-rolled at 1500°C in hydrogen. The actual rolling was done in a hot rolling mill with 20-inch wide rolls, and the zone refined bars were heated to 1500°C in a hydrogen furnace between each pass. The total reduction of the zone-refined tungsten bars was about 80 per cent to a finished thickness of 0.110 inch.

The evaluation of the sheet was all made at ARF. Microstructures and grain size were evaluated by standard techniques. Residual matrix contents were determined by wet chemical analyses. Elevated temperature tensile strengths were measured at different strain rates in argon up to 2000°C.

The equipment used in this series of experiments was all industrial (at Wah Chang), and it was concluded that the process is easily adaptable to a commercial operation.

III. RESULTS AND DISCUSSIONS

Pressing and sintering the 96 per cent tungsten composition presented no problems. The microstructure of the sintered alloy is reproduced in Figure 1.

The warm rolling (750°F) of sintered bars showed some interesting behavior. The alloy composition used in this investigation is a high-strength material. The strength reflected itself in the mill deflection during each pass. It was observed that, in general, the mill deflection was about 0.080 inch and increased somewhat as the roll gap was decreased. According to the operators, mill deflection of this order of magnitude was high but well within the capability of the mill used. Also, the warm rolling of the samples was stopped after reaching about 40 per cent reduction. This was thought to be the limit of reduction for this particular alloy, but in view

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of the fact that there was no evidence of edge cracking or surface checks, the piece probably could have been rolled more. This factor will be emphasized in the next series of experiments in that attempts will be made to roll the bars down as far as possible before zone refining.

The warm-rolled two-phase samples were about 3 inches wide and 0.4 inch thick. These were suspended by tantalum holders in the electron beam zone-refining furnace. Two samples were zone-refined at 2 inches per hour at 1900°C in vacuum. The hot zone was about 0.5 inch wide, and both the zone-refining experiments were completed with no difficulty. The life of the electron beam gun was not affected by these runs, implying thereby that no deposits of iron or nickel occurred on the electrode. This eliminated one of the major anticipated difficulties in this program. As stated before, the samples were suspended freely from the top and were carrying their own weights. It was established in previous work that this was not the best practice; hence, in the next series of experiments, samples will be held from the top and bottom--the reasons will become evident in discussions below.

The zone-refined samples were trimmed to remove the enriched zones, but no attempts were made to grind off the crown and unevenness in the surface. Then the samples were hot rolled at 1500°C with intermediate heating accomplished in hydrogen.

Sample A was hot rolled about 40 per cent before it cracked. The reductions per pass were 20 per cent. The reasons for this cracking were two-fold; the cracks initiated near the top end of the bar where the residual matrix was high (0.42) and the 20 per cent reductions per pass were quite severe. Sample C was hot rolled at the same temperature but with only 10 per cent reductions per pass. The rolling schedule is given in Table I. The mill deflections are not too large, as is evident from the data. Sample A was rolled down from 0.355 to 0.230 inch before it cracked in two. Sample C (Table I) was cross-rolled to a finished dimension of about 8 x 5 x 0.110

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inches. A photograph of this sample is shown in Figure 2.

The residual matrix content in both the sheets (Sample A and Sample C) was found to be about 0.13 to 0.16 weight per cent. The initial analysis mentioned in the last report was 0.06. But this was found to be due to an error in the chemical solution treatment which had not removed all the matrix. This error was first detected on the gaussmeter which will be discussed later. The residual matrix content was reasonably uniform throughout the thickness and length of the sample, the limits being 0.13-0.16 per cent. As far as the future work is concerned, this residual matrix can be brought down to 0.05 or less by initially warm rolling the sintered bar to 50 per cent (possibly more) and supporting the bar in the zone-refining furnace so that it need not carry its own weight. These factors will be kept in mind during the next series of experiments.

Some interesting effects were observed during hot rolling of the zone-refined bars. The residual matrix content of the edge of a sample (partially enriched) and the degree of reduction had a strong influence on the final microstructure, porosity, and grain size.

Figure 3 shows the microstructure of a section of Sample A which was hot rolled 20 per cent at 1500°C and found to have a residual matrix content of 0.28. The grain size has increased substantially over the liquid phase sintered alloy (Figure 1), and the pores are distributed mostly along grain boundaries. Another section of the sample treated identically but which had a residual matrix content of 0.2 (because it was farther from the rich end) shows almost no porosity and a recrystallized fine grain structure (Figure 4). Figures 5 and 6 show the fine grain microstructure of Sample C hot-rolled sheet in directions parallel and perpendicular to the direction of rolling.

The tensile strength measurements were made on Sample C hot-rolled sheet. The measurements were all made at 2000°C in argon. These were all made at three different strain rates on samples of sheet cut from directions

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parallel and transverse to rolling direction. The results are all summarized in Table II. The results indicate that the high-temperature strength values of the hot-rolled sheet are comparable to values for commercial tungsten sheets.* The strain rates had similar effects. In all cases, however, the elongation was low--of the order of 6-8 per cent. There are two reasons for this; one is the relatively high residual matrix content, and the other is the fact that, in hot-rolled tungsten sheets, no appreciable amount of ductility can be induced without finish rolling down to 1100°C. The transition temperature of this sheet (Sample C) is very high (~ 500°C), and this is undoubtedly due to the residual matrix content.

Also during this report period, the resistance-heated zone-refining furnace at ARF was put into operation, and a piece 20 in. x 2 in. x 0.5 in. was zone refined at 1900°C in vacuum. This will be hot rolled and evaluated. No operational difficulties were encountered.

IV. CONCLUSIONS

- (1) The initial experiments in making tungsten sheet by the new process were successful. No major difficulties were encountered in any step of the process. The whole process looks very promising insofar as industrial adaptation is concerned.
- (2) The initial steps of warm rolling of the liquid phase sintered alloys and zone refining conditions will govern the final properties of the hot-rolled sheet.
- (3) Although the high-temperature tensile properties of the sheets made thus far are comparable to those of commercial sheets, the residual matrix content should be lowered to around 0.05 per cent in order to obtain formable sheet.

* Union Carbide Metals Company, WADD TR 60-114, under Contract No. AF 33(616)-5600, Project No. 7351; and NASA TN D-79, September 1959.

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- (4) The gun life of the electron beam furnace was not the least bit affected by the zone refining of the sintered and warm-rolled bars.
- (5) The sheets obtained so far were texture-free insofar as their mechanical properties were concerned.

V. FUTURE WORK

- (1) Scale-up experiments are planned early in November when the writer will go out to Wah Chang Corporation in Albany, Oregon, in order to make 11-12 inch wide sheet. Some 11 x 11 x 1 inch plates have already been prepared by compacting in hydrostatic press and sintering.
- (2) Several factors will be investigated with regard to warm rolling. Attempts will be made to warm roll (750°-1000°F) sintered plates more than 50 per cent in order to obtain sheets which will be larger and closer to finished thicknesses.
- (3) Zone refining will be accomplished in such a manner as to remove all the load from the sample. This will be done by supporting the plates (vertically) from the top as well as the bottom.
- (4) Hot rolling on zone-refined plates will be carried out down to less than 0.100 inch thickness. These experiments will be performed on plates which will be cleaned up (trimmed and crown removed) soon after zone refining.
- (5) Work will continue on calibration of a gaussmeter used for rapid determination of residual matrix content. This is a very sensitive instrument, and factors such as probe gap, sample thickness, and temperature will have to be standardized before it can be used effectively.

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VI. LOGBOOKS AND CONTRIBUTING PERSONNEL

The data are recorded in ARF Logbook Nos. C-1180, and C-1200. R. Moll (Assistant Experimentalist), R. Hodson (Assistant Metallurgist), and N. M. Parikh contributed to this program. The personnel at Wah Chang Corporation who contributed to this program were Dr. James Wong (Director of Research) and Mr. A. Reison.

VII. RATE OF EFFORT

It is estimated that about 30 per cent of the work is completed.

Respectfully submitted,

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N. M. Parikh.

N. M. Parikh
Senior Metallurgist

NMP/cs

Tech. Rev. - DWL

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TABLE I

ROLLING SCHEDULE FOR HOT ROLLING SAMPLE C

(1500°C, Hydrogen)

Sample Thickness, inches	Roll Gap, inches	Remarks
0.420	0.350	Little edge cracking at top end.
0.380	0.300	No further edge cracking.
0.338	0.265	
0.309	0.235	
0.260	0.205	
0.240	0.170	
0.213	0.140	Crack 1 1/2 in. from bottom.
0.193	0.110	
0.173	0.080	
0.142	0.050	
0.125	0.050	
0.109	-	

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TABLE II

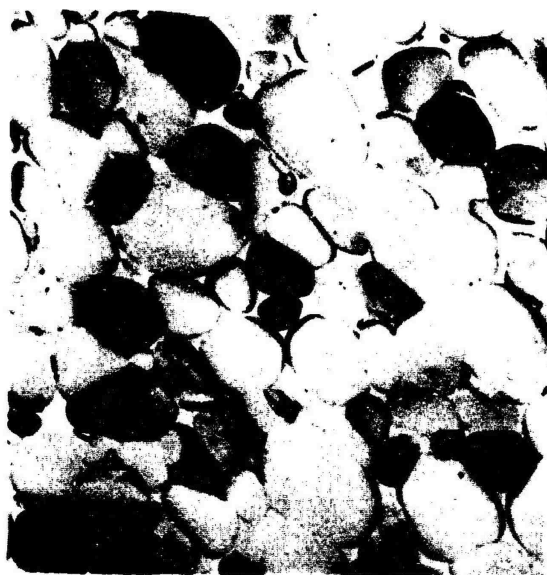
2000°C TENSILE STRENGTH OF SAMPLES
CUT FROM SHEET (SAMPLE C)
MADE BY THE ZONE-REFINING PROCESS

Specimen Direction *	Strain Rate, in/min	Tensile Strength, psi	Tensile Elongation, %
Parallel	0.062	8200	5
Transverse	0.062	8400	6
Parallel	0.8	11,200	7
Transverse	0.8	10,800	5
Parallel	2.0	14,500	8
Transverse	2.0	14,000	8

* Indicates the direction parallel or transverse to direction of rolling.

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Neg. No. 22012

250X

Figure 1

Microstructure of 96W-2.8Ni-1.2Fe alloy sintered at 1530°C in hydrogen for 2 hours. This was the starting material for the sheets made on this program.

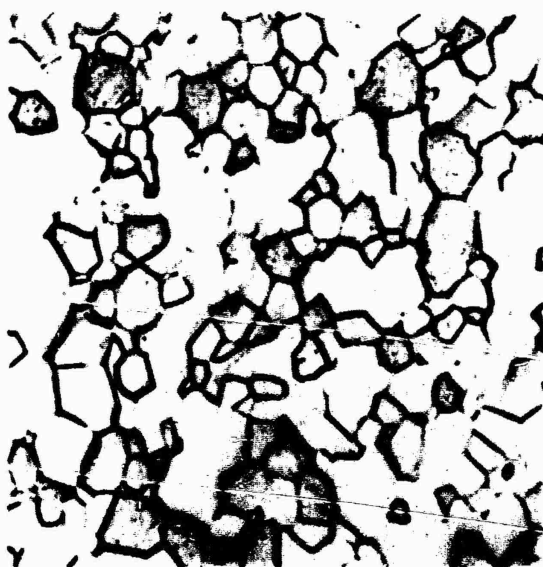


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Figure 3

Edge of sample containing 0.28 per cent residual matrix hot rolled 20 per cent. Note large grain size and porosity.

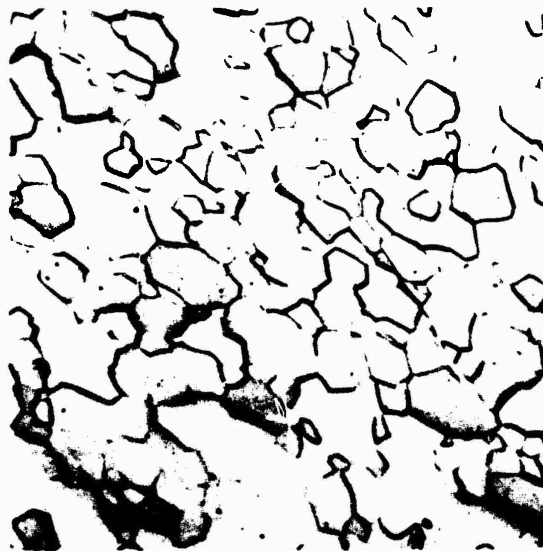


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Figure 4

Edge of sample containing 0.2 per cent residual matrix hot rolled 40 per cent. Note absence of porosity and fine grain size compared to one above.



Neg. No. 22019

250X

Figure 5

Sample containing 0.13 residual matrix content hot rolled about 80 per cent. Microstructure in the direction of rolling. Note fine grain size.



Neg. No. 22017

250X

Figure 6

Sample containing 0.13 residual matrix content hot rolled about 80 per cent. Microstructure transverse to rolling direction.

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Watertown Arsenal Laboratories, Watertown 72, Mass. ZONE REFINING OF CEMENTED TUNGSTEN-BASE ALLOYS - N. M. Parikh

Report No. WAL TR 372/32-1-TL, 17 pp - tables - illus, D/A Project 5B93-32-002, ORD Project TB4-002, Contract No. DA-11-022-505-ORD-3092, October 1961, Unclassified Report

This program concerns the development of a process for fabrication of tungsten sheet using tungsten-nickel-iron alloys as a starting material. Initial attempts in scaling up the process were successful.

(over)

Sintering, warm rolling and zone refining tungsten-nickel alloys presented no difficulties. Eight inch wide hot rolled tungsten sheets were made by this process.

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Watertown Arsenal Laboratories, Watertown 72, Mass. COPPER-COOLED TUNGSTEN FIBER ROCKET NOZZLE MATERIAL - T. J. Koppelman

Report No. WAL TR 372/32-2-T3, 17 pp - tables - illus, D/A Project 5893-32-002, ORD Project TB4-002, Contract No. DA-11-022-505-ORD-3092, October 1961. Unclassified Report

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